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(REV. 1-98)		ATTORNEY'S DOCKSTANIMBER 7.				
TRANSMITTAL LETTER		0020-4621P				
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INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED				
PCT/JP98/01692	14 April 1998 -	15 April 1997				
TITLE OF INVENTION						
CURING COMPOSITION APPLICANT(S) FOR DO/EO/US	OF FLUORORUBER AND MOLDED ARTIC	CLE OF FLUORORUBBER				
MA	TSUMOTO, Koji; SHIRAI, Yoshihi:	ro - A a				
Applicant herewith submits to the United States	Designated/Elected Office (DO/EO/US) the foll	owing items and other information:				
<ol> <li>This is a FIRST submission of items conc</li> </ol>	erning a filing under 35 U.S.C. 371.	(i, a, j)				
<ol><li>This is a SECOND or SUBSEQUENT su</li></ol>	bmission of items concerning a filing under 35 U.S	i.C. 371.				
	examination procedures (35 U.S.C. 371(f)) at					
	applicable time limit set in 35 U.S.C. 371(b)					
. ===	eliminary Examination was made by the 19 <sup>th</sup> n	nonth from the earliest claimed priority date				
5. A copy of the International Applicatio						
	ed only if not transmitted by the International	Bureau).				
b. has been transmitted by the Int		es - (DOME)				
c. is not required, as the application was filed in the United States Receiving Office (RO/US).						
X   A translation of the International Application into English (35 U.S.C. 371(c)(3)).   X   Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(2)).						
a. are transmitted herewith (required only if not transmitted by the International Bureau).						
a. are transmitted nerewith (required only if not transmitted by the international Bureau).  b. have been transmitted by the International Bureau.						
b. have been transmitted by the International Bureau.  c. have not been made; however, the time limit for making such amendments has NOT expired.						
d. have not been made and will n	<del>-</del>	and the transfer of the transf				
	he claims under PCT Article 19 (35 U.S.C. 37	/1(c)(3)).				
9. An oath or declaration of the invento	r(s) (35 U.S.C. 371(c)(4)).					
10. A translation of the annexes to the Ir	ternational Preliminary Examination Report u	under PCT Article 36				
(35 U.S.C. 371(c)(5)).						
Items 11. to 16. below concern document(s	or information included:					
11. An Information Disclosure Statemer	t under 37 CFR 1.97 and 1.98./International S	Search Report with cited references				
12. An assignment document for record	ng. A separate cover sheet in compliance with	1 37 CFR 3.28 and 3.31 is included.				
13. A FIRST preliminary amendment.						
A SECOND or SUBSEQUENT prel	iminary amendment.					
	,					
14. A substitute specification.						
15. A change of power of attorney and/o	or address letter.					
16. Other items or information: 1. Preliminary Amendment w/Ame	nded Claims					
2. Zero (0) Sheets of Formal Drawin	gs					

U.S. APPLICATION NO (IF known, see	9/403224 TITERNA	TIONAL APPLICATION NO	514 MGC	OF.	ATTORNEY'S DOC	KET NUMBER
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and International Se	arch Report not prepared by the	e EPO or JPO	. \$970.00			
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but international sear	rch fee (37 CFR 1.445(a)(2)) pa	nid to USPTO	. \$760.00	l .		
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International prelimi	nary examination fee (37 CFR	1.482) paid to USPTO				
but all claims did not	satisfy provisions of PCT Arti	cle 33(1)-(4)	. \$670.00			
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and all claims satisfie	ed provisions of PC1 Article 3:	3(1)-(4)	. \$96.00			
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CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE			
Total Claims	6 - 20 =		X \$18.00	\$		
Independent Claims	1 - 3 =		X \$78.00	s		
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> PATENT 0020-4621P

IN THE U.S. PATENT AND TRADEMARK OFFICE

Applicant:

MATSUMOTO, Koji et al

- - -

Int'l. Appl. No.: PCT/JP98/01692

Appl. No.:

NEW

Group:

Filed:

October 15, 1999

Examiner:

For:

CURING COMPOSITION OF FLUORORUBBER AND MOLDED ARTICLE OF FLUORORUBBER

## PRELIMINARY AMENDMENT

### BOX PATENT APPLICATION

Assistant Commissioner for Patents Washington, DC 20231

October 15, 1999

Sir:

The following Preliminary Amendments and Remarks are respectfully submitted in connection with the above-identified application.

## AMENDMENTS

# IN THE SPECIFICATION:

Please amend the specification as follows:

Before line 1, insert --This application is the national phase under 35 U.S.C. § 371 of PCT International Application No. PCT/JP98/01692 which has an International filing date of April 14, 1998, which designated the United States of America.--

### REMARKS

The specification has been amended to provide a crossreference to the previously filed International Application.

If necessary, the Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit Account No. 02-2448 for any additional fees required under 37 C.F.R. § 1.16 or under 37 C.F.R. § 1.17; particularly, extension of time fees.

Respectfully submitted,

BIRCH, STEWART, KOLASCH & BIRCH, LLP

By wallell

ANDREW D. MEIKLE Reg. # 32,868

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(Rev. 03/30/99)

#### SPECIFICATION

CURING COMPOSITION OF FLUORORUBBER AND MOLDED ARTICLE OF FLUORORUBBER

#### 5 FIELD OF THE INVENTION

The present invention relates to a curing composition of a fluororubber, and a molded article of a fluororubber. In particular, the present invention relates to a curable composition of a fluororubber, which provides a molded article having practically sufficient properties, for example, heat-resistance, without secondary curing, and a molded article of a fluororubber which is produced by molding and curing such a composition.

BACKGROUND ART

Fluororubbers are used as industrial materials in a wide
15 variety of technical fields, since they have much better
heat-resistance, oil-resistance, solvent-resistance,
chemical-resistance, etc. than general-purpose rubbers.

Fluororubbers are often used under severe conditions such as a temperature of 200°C by making use of their particularly good heat-resistance. However, for achieving heat-resistance under such severe conditions, it is inevitable to mold the curable composition of fluororubbers, subjecting the molded article to primary curing and then subjecting the cured article to secondary curing to complete crosslinking and to release gasses generated in the course of crosslinking.

## SUMMARY OF THE INVENTION

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However, a curing composition comprising a fluororubber, which provides a molded article having practically sufficient

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properties only by primary curing without secondary curing, has been sought, since the secondary curing requires a large amount of heat energy and additional work.

Thus, one object of the present invention is to provide such a curing composition of a fluororubber.

Accordingly, the present invention provides a curing composition of a fluororubber comprising a fluororubber which is curable with an organic peroxide, a polyfunctional unsaturated compound, and an organic peroxide, wherein the total amount of acetone and tert.—butanol contained in the decomposed products of said organic peroxide, which are generated at a curing temperature, is 2 moles or less per one mole of the decomposed products.

DETAILED DESCRIPTION OF THE INVENTION

Fluororubbers contained in the composition of the present invention may be any known fluororubbers that can be cured with organic peroxides. Preferred examples of such fluororubbers are as follows:

Vinvlidene fluoride base fluororubbers:

VdF-HFP copolymers, VdF-HFP-TFE copolymers, VdF-PFP copolymers, VdF-PFP-TFE copolymers, VdF-PEMVE-TFE copolymers, VdF-PFMVE-HFP copolymers, VdF-CTFE copolymers, VdF-HFP-E copolymers, VdF-HFP-TFE-E copolymers

 $\label{eq:the_problem} \mbox{The abbreviations used in the above have the following} $$25$ meanings:$ 

VdF: Vinylidene fluoride

HFP: Hexafluoropropylene

TFE: Tetrafluoroethylene

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PFP: Pentafluoroprpylene

PFMVE: Perfluoro (methyl vinyl ether)

CTFE: Chlorotrifluoroethylene

E: Ethvlene

5 Propylene-tetrafluoroethylene fluororubbers:

Propylene (30-60 mole %)-tetrafluoroethylene (40-70 mole %) copolymers

Such copolymers may comprise 0 to 20 mole % of one or more other monomers which can be copolymerizable therewith.

 $\label{thm:condition} \mbox{Tetrafluoroethylene-perfluoro(alkyl vinyl ether)} \\ \mbox{copolymers:}$ 

Tetrafluoroethylene (40-85 mole %)-perfluoro(alkyl vinyl ether) (15-60 mole %) copolymers

Other fluororubbers:

 $\label{prop:licone} \mbox{Fluorosilicone rubbers, polyfluoroalkoxyphosphazene} \\ \mbox{rubbers.}$ 

There are various methods to make fluororubbers curable with organic peroxides.

For example, fluororubbers are prepared by polymerizing monomers in the presence of iodine-containing compounds such as  $\mathrm{CH_2I_2}$ , or iodine/bromine-containing compounds such as  $\mathrm{CH_2IBr}$ , by copolymerizing diene compounds such as  $\mathrm{CF_2=CF-CF=CF_2}$ , or by heat treating prepared polymer to introduce double bonds in molecules.

Polyfunctional unsaturated compounds contained in the composition of the present invention may be ones that are known as curing aids. Preferred examples of polyfunctional unsaturated compounds are triallyl isocyanurate, trimethallyl isocyanurate, triallyl cyanurate, 
The amount of polyfunctional unsaturated compounds is from 0.1 to 10 wt. parts, preferably from 1 to 5 wt. parts, per 100 wt. parts of the fluororubbers.

Organic peroxides used according to the present invention

are such compounds that generate decomposed products containing
acetone and tert.-butanol in a total amount of 2 moles or less
per one mole of the decomposed products, when they are decomposed
under a curing temperature condition.

Table 1 summarizes the compositions of low-boiling

10 decomposed products of several known organic peroxides under curing temperature conditions.

Table 1

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Organic peroxide	Low-		decompos	ed products
berovide				
	Methane	Ethane	Acetone	tertbutanol
1	0.56	0.37	2.14	1.30
2	1.15	0	0	0
3	0.62	0	0.32	0.7
4	0.53	0	0.56	1.14

Notes 1: 2,5-Dimethyl-2,5-bis(tert.-butylperoxy)hexane
(Perhexa 25B)

2: Dicumyl peroxide (Percumyl D)

3: tert.-Butylcumyl peroxide (Perbutyl C)

4: di-tert.-Butyl peroxide (Perbutyl D)

Among the organic peroxides which generate decomposed products containing 2 mole/mole or less of acetone and tert-butanol in total, dicumyl peroxide is preferable, since it generates neither acetone nor tert.-butanol.

The amount of organic peroxides is from 0.3 to 1.2 wt. parts, preferably from 0.4 to 1.0 wt. parts, per 100 wt. parts of fluororubbers. When the amount of organic peroxides exceeds 1.2

wt. parts, the weight change in use increases, which may cause some practical problems.

The curing composition of fluororubbers according to the present invention may optionally contain conventional additives 5 which are compounded in fluororubbers, for example, fillers, processing aids, plasticizers, colorants, etc.

The curing composition of fluororubbers according to the present invention can provide cured molded articles, which have practically sufficient properties and in which the contribution of secondary curing to a compression set (which will be defined below) is 30 % or less, only by primary curing which is applied to the conventional curing compositions of fluororubbers.

Curing conditions may be the same as those used to cure the conventional curing compositions of fluororubbers. For example, the compositions are cured for 0.1 to 1 hours at a curing temperature in a range between 150 and 190°C, under a curing pressure in a range between 1 and 10 Pa.

#### EXAMPLES

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Comparative Example 1

Medium thermal carbon (MT-C) (20 wt. parts), triallyl isocyanurate (TAIC-M60, 60 % diluted product of triallyl isocyanurate, manufactured by NIPPON KASEI KABUSHIKIKAISHA) (6.7 wt. parts) and Perhexa 25B (manufactured by NOF Corporation) (0.5 wt. part) were compounded in DAIEL G-912 (an iodine-containing fluororubber manufactured by DAIKIN INDUSTRIES, LTD.) (100 wt. parts), and well kneaded on open rolls to obtain a testing compound.

The obtained compound was molded, and subjected to primary

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curing at  $160^{\circ}$ C for 10 minutes, and secondary curing at  $180^{\circ}$ C for 4 hours to obtain a sheet ( $120 \text{ mm} \times 150 \text{ mm} \times 2 \text{ mm}$ ) for the measurement of physical properties, and a P-24 O-ring for the measurement of a compression set.

With a molded product which had been primarily cured and one which had been primarily and secondarily cured, the following measurements were carried out.

 $\rm M_{100}$  (100 % modulus),  $\rm T_s$  (tensile strength at break) and  $\rm E_8$  (elongation at break) of a sheet were measured according to JIS K6301, and Hs (hardness) of a sheet was measured according to JIS K6253, Type A.

ΔW was a weight change rate expressed by the formula:

[(weight of secondarily cured product - weight of primarily cured product)] x 100 %

CS (compression set) was measured using a P-24 O-ring, which is defined by JIS B2401, under the conditions of 200°C, 70 hours and 25 % compression, according to JIS K6301.

The contribution of secondary curing to a compression set was evaluated by the formula:

20 [(CS, - CS,)/CS,] x 100 %

in which  $CS_1$  is the compression set of a primarily cured product, and  $CS_2$  is the compression set of a secondarily cured product.

Comparative Examples 2 and 3

A composition was prepared, a sheet and an O-ring were molded and then their properties were measured by the same methods as in Comparative Example 1 except that the amount of Perhexa 25B was changed to 1.0 wt. part or 1.5 wt. parts.

Comparative Examples 4 and 5

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A composition was prepared, a sheet and an O-ring were molded and then their properties were measured by the same methods as in Comparative Example 1 except that 0.25 wt. part or 1.5 wt. parts of Percumyl D (manufactured by NOF Corporation) was used in place of Perhexa 25B.

Comparative Example 6

A composition was prepared, a sheet and an O-ring were molded and then their properties were measured by the same methods as in Comparative Example 1 except that DAIEL G-902 (an iodine-containing fluororubber manufactured by DAIKIN INDUSTRIES, LTD.) was used as a fluororubber.

Comparative Example 7

A composition was prepared, a sheet and an O-ring were molded and then their properties were measured by the same methods as in Comparative Example 1 except that DAIEL G-902 (an iodine-containing fluororubber manufactured by DAIKIN INDUSTRIES, LTD.) was used as a fluororubber, and the amount of Perhexa 25B was changed to 1.5 wt. parts.

Examples 1 and 2

A composition was prepared, a sheet and an O-ring were molded and then their properties were measured by the same methods as in Comparative Example 1 except that 0.5 wt. part or 1.0 wt. part of Percumyl D was used in place of Perhexa 25B.

Example 3

A composition was prepared, a sheet and an O-ring were molded and then their properties were measured by the same methods as in Comparative Example 1 except that 1.0 wt. part of Perbutyl C was used in place of Perhexa 25B.

Example 4

A composition was prepared, a sheet and an O-ring were molded and then their properties were measured by the same methods as in Comparative Example 1 except that 1.0 wt. part of Perbutyl D was used in place of Perhexa 25B.

Example 5

A composition was prepared, a sheet and an O-ring were molded and then their properties were measured by the same methods as in Comparative Example 1 except that DAIEL G-902 (an iodine-containing fluororubber manufactured by DAIKIN INDUSTRIES, LTD.) was used as a fluororubber, and 0.5 wt. part of Percumyl D was used in place of Perhexa 25B.

The results are shown in Table 2.

TOUTON THUMBERD

Table 2									0	Me lam		
		U	Comparative Example No.	ive Exan	uple No.				X I	Example No.		
	-1	2	3	4	2	9	7	-1	2	0	4	ro l
Composition	100	100	100	100	100	1	1	100	100	100	100	1
-DATEL G-902			1		1	100	100	-	-		-	100
1000 O TILLE	20	20	20	20	20	20	20	20	20	20	20	20
-TAIC M60	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7
-Perhexa 25B	0.5	1.0	1.5	1	1	0.5	1.5	ł	-	!		1
-Percumyl D	1	1	!	0.25	1.5	1	!	0.5	1.0	-	1	0.5
-Perbutyl C	1	-	1	1	1	1	ł	1	1	1.0		
-Perbutyl D	1				1	1		:		:	1.0	:
Mechanical properties												
aft. primary curing												
(160°C x 10 min.)												
-M100	112	119	114	62	115	37	40	119	100	128	131	χ. Σ.
-T <sub>B</sub>	205	216	186	166	195	180	206	211	195	225	22./	184
E E	175	170	170	240	170	350	310	175	170	175	165	320
SH H	74	75	73	73	73	72	71	75	74	74	75	72
-CS (200°C x 70 hrs)	29.5	24.9	25.0	37.0	24.0	27	30.0	21.8	21.2	23.9	23.3	23
Mechanical properties												
aft. secondary curing												
(180°x 4 hrs.)												
-M100	152	147	154	68	151	46	51	148	135	164	153	49
· -	273	252	284	228	248	230	254	253	264	275	276	221
1 (s)	175	165	160	210	160	300	280	165	160	165	165	300
	77	77	77	74	77	72	74	77	9/	77	77	72
-cs (200°C x 70 hrs)	20.4	17.3	17.1	18.9	17.3	23.0	28.0	19.0	16,9	19.1	18.0	21
-∆W (%)	0.44	0.85	1.20	0.42	1.13			0.51	0.42	0.76	0.37	
[(CS <sub>1</sub> -CS <sub>2</sub> )/CS <sub>2</sub> )]x100	44.6	43.9	46.2	95.8	38.7	17.4	7.1	14.7	25.4	25.1	29.4	9.5

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As can be understood from the results of Examples 1-5, the compositions of the present inventions provide O-rings having a small compression set  $(200^{\circ}\text{C} \times 70 \text{ hours})$  only by primary curing.

As can be seen from the results of Comparative Example 4, the use of dicumyl peroxide in an amount of 0.25 wt. part deteriorates a compression set, since the curing does not sufficiently proceed. The results of Comparative Example 5 indicates that the use of dicumyl peroxide in an amount of 1.5 wt. parts significantly increases the weight change  $\Delta W$  (%).

Molded articles, which are produced by curing the compositions of the present invention, have less contribution of secondary curing to a compression set, and achieve a smaller compression set than those produced from conventional compositions after the primary curing, when the same fluororubbers are used. Thus, it is understood that molded articles produced from the compositions of the present invention have good practical usefulness without being secondarily cured.

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#### CLATMS

- A curing composition of a fluororubber comprising 100 parts by weight of a fluororubber which is curable with an organic peroxide.
- 0.1 to 10 parts by weight of a polyfunctional unsaturated compound, and
- 0.3 to 1.2 parts by weight of an organic peroxide, wherein the total amount of acetone and tert.-butanol contained in the decomposed products of said organic peroxide, which are generated at a curing temperature, is 2 moles or less per one mole of the decomposed products.
- 2. A curing composition according to claim 1, wherein said fluororubber is an iodine-containing fluororubber containing 0.01 to 5 % by weight of iodine atoms.
- A curing composition according to claim 1, wherein said organic peroxide is dicumyl peroxide.
  - 4. A curing composition according to any one of claims 1 to 3, wherein the amount of said organic peroxide is from 0.4 to 1.0 parts by weight per 100 parts by weight of said fluororubber.
- 5. A molded article of a fluororubber comprising a cured material of a curing composition as claimed in claim 1.
  - 6. A molded article according to claim 5, wherein the contribution of secondary curing to a compression set defined by the following formula is 30 % or less:
- 25 [(CS<sub>1</sub> CS<sub>2</sub>)/CS<sub>2</sub>] x 100 %.

in which  $CS_1$  is the compression set of a primarily cured product, and  $CS_2$  is the compression set of a secondarily cured product.

### ABSTRACT

A curing composition of a fluororubber comprising a fluororubber which is curable with an organic peroxide, for example, an iodine-containing fluororubber, a polyfunctional unsaturated compound, and an organic peroxide, for example, dicumyl peroxide, in which the total amount of acetone and tert.-butanol contained in the decomposed products of the organic peroxide, which are generated at a curing temperature, is 2 moles or less per one mole of the decomposed products.

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PLEASE NOTE: YOUMUST COMPLETE THE FOLLOWING:

## COMBINED DECLARATION AND POWER OF ATTORNEY FOR PATENT AND DESIGN APPLICATIONS

As a below named inventor, I hereby declare that: my residence, post office address and citizenship are as stated next to my name; that I

	verily believe that I am the original, fit plural inventors are named below) of							
sert Title: →	CURING COMPOSITION	N OF FLUORORUBBE	R AND MOLDED ARTIC	LE OF				
	FLUORORUBBER							
Ill in Appropriate  formation —  or Use   //ithout pecification	the specification of which is attached the specification was fi United States Applicati and amended on	led on ion Number	(if appli	as; cable): and/or				
ttached:	the specification was filed			as PCT				
	International Application		01692 /	; and was				
	amended under PCT Article 19 on (if applicable)							
sen Priority	amended by any amendment referre I acknowledge the duty to disclos § 1.56.  I do not know and do not belie thereof, or patented or described in a to this application, that the same has application in any country foreign to more than twelve months (six month this invention has been filled in any cor assigns, except as follows.  I bereby claim foreign priority I inventor's certificate I sisted below at filling date before that of the applica Prior Foreign Application!	It to above, se information which is material to we the same was ever known or us ny printed publication in any count as not in public use or on sale in not been patented or made the sub the United States of America on s for designs) prior to this applicat unity foreign to the United States of connectification of the printed States of properties and the state of the printed States of printed States printed br>printed prin	of the above identified specification patentability as defined in Title 37, Cc sed in the United States of America be try before my or our invention thereof the United States of America more their of the third States of America more their of the or my legal distributions of their or my legal ton, and that no application for patent America prior to this application by me tes Code, \$119 (a)-(d) of any foreign foreign application for patent or invention of the code of the code of the code of the code of their or invention of the code of the co	de of Federal Regulations, efore my or our invention or more than one year prior han one year prior to this de before the date of this representatives or assigns or inventor's certificate on or my legal representatives application(s) for patent or entor's certificate having a  Priority Claimed				
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	(Filing Date) (Application Number) (Filing Date) (Application Number) (Filing Date) (Applications, if any, for any Patent or Inventor's Certificate Filed More than 12 Months (6 Months for Designs) Prior to the Filing Date of This Application:							
nsert Requested information:	Country	Application I	Number Date of F	Filing (Month / Day / Year)				
	insofar as the subject matter of each the manner provided by the first par	of the claims of this application is agraph of Title 35, United States C d in Title 37, Code of Federal Regi	of any United States and/or PCT appling the disclosed in the prior United States Code, §112, I acknowledge the duty to ulations, §1.56 which became available of this amplication:	s and/or PCT application in disclose information which				
nsert Prior U.S. Application(s):	F SFF		. F					
if any)	(Application Number)	(Filing Date	(Status — pate	ented, pending, abandoned)				

(Filing Date)

(Status --- patented, pending, abandoned)

(Application Number)

I hereby appoint the following attorneys to prosecute this application and/or an international application based on this application and for transact all the first states and fractorists (cited to the context therewith and in connection with the resulting patent based on instructions received northindentially with international application papers to the attorneys identified below, unless the inventor(s) or assignee provides said attorneys with a written notice to the contrary:

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PLEASE NOTE: YOUMUST COMPLETE THE FOLLOWING:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

i	false statements may jeopare	dize the validity of the applic	ation or any patent issued thereon.				
ull Name of First or Sole Inventor;	GIVEN NAME	FAMILY NAME	INVENTOR'S SIGNATURE		DATE*		
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